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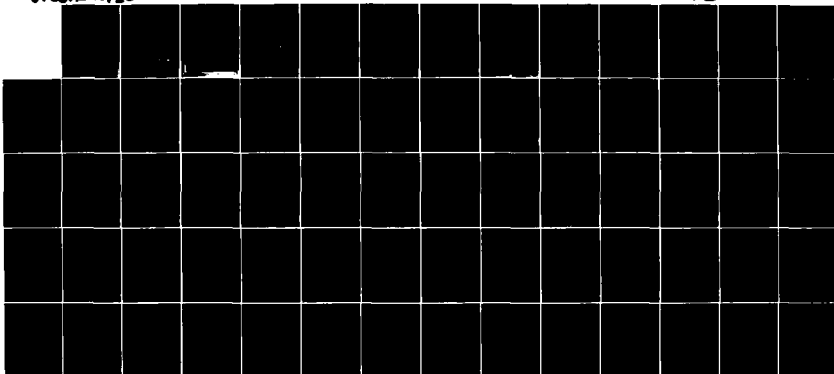
HEAVY METAL UPTAKE BY C ESCULENTUS S ALTERNIFLORA AND  
AGRONOMIC PLANTS FR. (U) UNIVERSITY COLL OF WALES  
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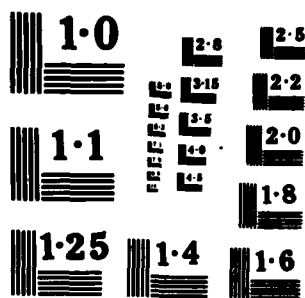
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HEAVY METAL UPTAKE BY *C. esculentus*, *S. alterniflora* AND AGRONOMIC  
PLANTS FROM CONTAMINATED SOILS AND SEDIMENTS

PRINCIPAL INVESTIGATOR  
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**AD-A161 566**

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CONTRACT NUMBER: DAJA37-82-C-0195

FIRST PERIODIC REPORT  
February 2, 1982 through February 28, 1982.

The research reported in this document has been made possible through  
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## FIRST INTERIM REPORT

The contract commenced on February 2, 1982. Miss N.J. Houghton has been appointed for a 24 month contract on scale 1b of British university scientific staff. Miss Houghton is an honours graduate in Environmental Science from Aberystwyth. At present she is preparing to submit her Master's dissertation on selenium and other trace elements in soils and plants.

Immediate action has been taken to initiate the experimental work concerned with growing *C. esculentus* and agronomic plants in a glasshouse. It is proposed to collect soils from these localities:-

- 1) University farmland (Control)
- 2) Ystwyth valley (High Pb, medium Zn, low Cu and Cd)
- 3) Halkyn Mountain (High Pb, Zn, low Cu and medium Cd)
- 4) Shipham (High Pb, Zn and Cd, low Cu)
- 5) Tamar Valley (Medium Pb, low Zn, High Cu, low Cd)

Sites 1, 2 and 3 have been visited and soil samples collected; sites 4 and 5 will be visited before the end of February. The results from analysing these soils will be used to make a final selection for the growth experiment. Sufficient quantities of each soil will then be collected in March and potted in April. Seed sowing will take place in mid-May.

The species sown will include a grass, wheat, lettuce and radish and *C. esculentus*. Treatments will be triplicated and one set of soils will be sown without amendment, the otherset will be limed, as necessary, to raise pH to 6.5 - 7.0. Fertilizer will also be added (NPK) amounts to be decided following a major nutrient analysis of the soils. The experimental procedure for the flooded soils (*C. esculentus*) will be agreed with WES personnel.

Consultations and investigations are in hand to locate contaminated marine and estuarine sediments. Planning of the saltwater experiment will follow.

HEAVY METAL UPTAKE BY C. esculentus, S. alterniflora AND AGRONOMIC PLANTS  
FROM CONTAMINATED SOILS AND SEDIMENTS

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CONTRACT NUMBER: DAJA37 - 82 - C - 0195

SECOND INTERIM REPORT

February 1, 1982 through July 31, 1982



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## INTRODUCTION

The First Interim Report was concerned with the appointment of staff and the initiation of the contract. This Second Interim Report is concerned with the setting up of the field and greenhouse experiments.

Dr. B.E. Davies and Ms. N.J. Houghton visited the Waterways Experiment Station at Vicksburg, Ms. in April 1982. Three working days were spent reviewing the experimental program with Dr. C.R. Lee and gaining familiarity with the WES methodology.

Dr. C.R. Lee visited Aberystwyth in June 1982 to continue collaboration with Dr. Davies and Ms. Houghton. Dr. Lee brought with him young plants of Spartina alterniflora and Cyperus esculentus. These were imported under MAFF licence PHF 461/9 and USDA Phytosanitary Certificate A315612.

Tensiometers, double buckets and associated apparatus was imported from WES in May 1982. Supplies of the WES reference soil were imported under MAFF licence.

## AGRONOMIC PLANTS AND FRESHWATER (FLOODED) EXPERIMENT

An initial sampling of 60 locations in 6 areas was made and the samples were analysed: this allowed the selection of 5 experimental soils comprising a range of heavy metal contents. Analytical data for the selected soils are:-

AREA	pH	Lime Requirement (cwt/acre)	EDTA extract: µg/g soil			
			Pb	Zn	Cu	Cd
Frongoch	6.6	-	21	30	13	0.4
Ystwyth Valley	5.8	66	1500	65	12	0.7
Halkyn Mountain	5.7	60	1525	1625	32	0.7
Somerset	7.2	-	1250	3450	6	115
Parys Mountain	5.9	15	365	19	550	0.4

Frongoch is one of the U.C.W. farms and is the local control soil. Ystwyth valley is a Pb-contaminated site. Halkyn Mountain, Clwys, is high Pb, Zn. Somerset (i.e., Shipham) is high Pb, Zn, Cd. Parys Mountain is an old open-pit copper mine and has high Cu, medium Pb.

The selected soils were air-dried, hand screened to remove larger debris and mixed. The soil was then mixed with coarse gravel (1 pt gravel : 4 pt soil) to improve drainage and placed above glass fiber and 2.5 cm of sand in 9 inch plastic pots to within 2.5 cm of the top. A second portion of each experimental soil was potted in the same way but, prior to potting, lime ( $\text{CaCO}_3$ , commercial) was added to each soil. Thus, there were two basic treatments - unlined and limed with the exception of the Somerset soil the pH of which was 7.2 and therefore liming was not required.

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The agronomic plants selected were radish (Webb's French Breakfast), lettuce (Paris White), wheat (spring), red fescue (Merlin) and ryegrass (S24). Each treatment was replicated three times. The pots were watered (tap water) twice daily for three days and then fertilized. To each pot a basal fertilizer solution was added.

After fertilization the pots were allowed to equilibrate for one week. They were sown (June 10, 1982) by scattering seed profusely across the soil surface. The pots were then watered twice daily. After germination those pots where rapid growth had taken place were thinned on June 20th. All pots, except wheat, were again thinned on June 28th. The first ryegrass cut (15 cm growth) was on July 1st. Wheat was thinned on July 5th. Radish, lettuce and ryegrass were moved out of doors on July 1st.

Cyperus esculentus seedlings were transplanted on June 22nd to the five contaminated soils (27 pots) and the WES reference soil (3 pots) : these were all in standard, plastic 9 inch pots. In order to check how the smaller rooting volume in these pots compared with the inner pots of the double bucket assemblage a further set of inner bucket pots was prepared : 3 each of Halkyn, Ystwyth and WES and 2 of Parys Mountain. Tensiometers were inserted in these pots with the intention that watering take place at the 50 bar meter reading.

Cyperus esculentus seedlings were also transplanted into the double bucket assemblages containing the 5 contaminated soils (27) and the WES soil (3 x replication). In the flooded replicates 5 cm depth of water is maintained above the soil surface.

In this part of the experiment (flooded and non-flooded) the limed/unlimed plus basal fertilizer treatments were maintained. The WES soil was fertilized and, as appropriate, limed. To each WES pot was added.

	$(\text{NH}_4)_2 \text{SO}_4$	2.17 g
	$\text{NaH}_2\text{PO}_4$	2.11 g
	KCl	0.88 g
and	$\text{CaCO}_3$	25.3 g

SPARTINA : GREENHOUSE AND FIELD

Fourteen estuarine and salt marsh sediments were collected from the Tamar and Tavy Estuary, the Avon Estuary, the Usk (tidal), Llanelli Docks, the Loughor Estuary, the Clarach and Leri estuaries and the Conwy estuary. The following five sediments were selected.

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Total metal : ( /g

	Pb	Zn	Cu	Cd
1. Tamar St.D.	99	147	159	1.3
1. Tavy B.F.	127	180	149	0.9
2. Clarach - Llangorwen	1103	30	67	0.3
2. Conwy - L.	93	61	14	0.7
1. Avon - NW.	43	92	17	0.5

1. West of England
2. Wales

Sufficient inner buckets were filled with sediment at the field site and returned to Aberystwyth. Inner buckets were also filled with sediment from the Dyfi estuary, the planned location of the field experiment.

In the greenhouse replication was 4 X and no fertilizer liming treatment was included. A further 4 pots were filled with the WES soil.

Spartina alterniflora seedlings were transplanted on June 22 with a planned harvest date of October 22 (120 day growth period). Initially, the pots were kept flooded with tap water until a good establishment was observed. Subsequently this water is to be replaced by synthetic seawater ('Instant Ocean') imported from WES.

For the field experiment the procedure followed that above. Once it was clear that the seedlings were viable the pots (inner buckets) were transported to the Dyfi estuary and embedded in the native Spartina marsh. The pots were fitted with nylon mesh protective covers according to standard WES procedure.

#### PROSPECTIVE

Between this report date and the next (January 1983) the plants will be harvested as appropriate. The natural Spartina marsh of the source area will be sampled. The Dyfi estuary pots will be reclaimed but kept intact and viable for future work. An analytical schedule has been agreed with WES.

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Heavy metal uptake by Cyperus esculentus, Spartina alterniflora  
and Agronomic crops from contaminated soils and sediments

R+D-429-REN

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Third interim report

July 1982 through January 1983

CONTRACT NUMBER DAJA37 - 82 - C - 0195

The research in this document has been made possible through the support and sponsorship of the U.S Army. This report is intended only for the internal management use of the contractor and the U.S Govt.

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# **1 AGRONOMIC PLANTS AND FRESHWATER (FLOODED) EXPERIMENT**

## **1.1 AGRONOMIC PLANTS**

As specified in the second interim report, the radish, lettuce and grass experiments were moved outside on July 1st. The management regime of these and the other plants is presented in Table 1.

### **1.1.1 RADISH AND LETTUCE**

All four species were placed on polythene sheeting in the outdoor environment. A daily watering regime was maintained throughout the growing period at about 4pm, for 30-45 minutes, and the plants randomised around a central sprinkler, such that each pot received the same amount of water daily.

Rapid growth was observed in both radish and lettuce, and all pots were harvested on July 13th when the plants were at their optimum growth stage. Following harvest, the radish were divided into bulbs and leaves, and fresh weight recorded. The fresh weights of all above ground lettuce parts were also recorded. All parts were then subjected to the standard washing procedure i.e washed twice in tapwater, and finally in distilled water. Surface dirt from the radish bulbs was removed with a nylon nail brush, and the bulbs diced with a stainless steel knife to assist drying. The plants were then placed in paper bags and dried at 100°C in an oven with a forced draught.

Most of the plant material dried within 24 hours, that which contained more fibrous tissue occasionally took longer. Oven dry weights were recorded immediately (Tables 2 & 3), and the moisture contents calculated (Tables 2 & 3).

### 1.1.2 Grass

At first, both grass species showed rapid growth, the ryegrass more so than the Festuca. Maintaining the 15cm harvest height, 4 cuts were taken from the ryegrass between moving the pots outdoors and Aug 12th, and 3 from the Festuca. By this time growth had slowed considerably. A final harvest was made on October 27th, before the plants were left for the winter period. Little growth has occurred since this date.

All cuts were subjected to the washing and drying regime, following fresh weight recordings, dried in the same manner as the vegetables, and dry weights recorded. All are shown in Tables 4 & 5, along with moisture contents.

### 1.1.3 WHEAT

Following thinning on July 5th, saucers were placed under the pots, the assemblage placed on a table 3' high in the middle of the greenhouse and randomised. Temperature and relative humidity readings were recorded daily and means are presented in Table 6.

The plants grew rapidly, and a second thinning was required on July 22nd, by which time, differences between the trials were already visible. Fresh and dry weights, and height are presented in Table 7. A daily watering regime was maintained at about 4pm. Watering was from underneath, filling the saucers to ensure a plentiful supply.

Aphids were observed on some of the plants during July, and were thereafter restrained using a commercial pesticide, which had been previously analysed and found to contain no detectable traces of Pb, Zn Cu or Cd. Sprayings with fresh solution on alternate days were found to be sufficient.

Growth slowed during September, and the onset of ripening followed. Differences between the trials were, by now, distinct, and have been recorded on photographs.

By the beginning of October, after a growth period of 115 days, the ripening stage was complete, and the plants ready for harvest.

The above ground plant parts were harvested, their height measured, and divided into heads and stalks, both of which were weighed fresh, and subjected to the washing and drying regime. Fresh weights only were recorded; although the material was subsequently dried, further water loss, was negligible. All data are presented in Table 7.

## 1.2 CYPERUS ESCULENTUS

### 1.2.1 UPLAND

Tensiometers were inserted into all of these pots, and the plant watered from below when readings exceeded the 50 bar meter. Rapid growth resulted with the Cyperus plants soon occupying the entire pot.

Many of the plants flowered in early September, by which time some treatment response was observed.

### 1.2.2 FLOODED

Growth patterns in the flooded part of the experiment mirrored those of the upland plants, and were more marked in certain cases. Some plants flowered in early September, the majority following soon after.

The water in the outer buckets was changed weekly and replaced with fresh tapwater, the height of water above the inner buckets maintained at 5 cm. The buckets were also scrubbed periodically to

reduce the build-up of algal growth which collected in some pots.

#### 1.2.3 ALL POTS

During early August, 18 Eh probes were received from WES, and inserted into both flooded and upland pots (9 in each, 1 per treatment). Readings on alternate days in the upland pots, and weekly in the flooded, were taken after this date, and are presented in Tables 8 & 9.

From these tables (9), in the upland experiment, after the first two readings, all gave high positive Eh readings, verifying the oxidising nature of the environment, and these conditions were maintained at a reasonably consistent level throughout. The highest readings were observed in the Somerset and Parys Mountain soils, but all were between 300 and 600 mV. In the flooded experiment (Table 8), reduced conditions were maintained in all pots, except, curiously in the Halkyn soils (both treatments), which remained positive despite continuous flooding. Some of the Somerset readings behaved likewise, but for no apparent reason.

#### 1.2.4 HARVEST - UPLAND

As mentioned, flowering started at the beginning of September, and 78 percent of plants flowered in the upland experiment; the particular treatments which flowered are indicated (\*) in Table 10. vegetative growth had been reached and no new flowering was expected. The onset of senescence was thought to be imminent.

Plants were divided into;

Flower heads,

Above ground parts,

Roots, and weighed.

Flower heads were dried and stored for analysis individually.

The above ground plant parts and roots were washed and dried, particular care given to roots to remove persistent soil particles. Fresh and dry weights, and percentage water contents are given in Table 10.

#### 1.2.5 HARVEST - FLOODED

The proportion of plants, which flowered in this part of the experiment was 80%, the plants involved indicated (\*) in Table 11. Flower heads were removed and dried for later analysis. The plants were again divided into above ground parts and roots, and each washed thoroughly, weighed, dried and stored. Sediments were replaced in the inner buckets, and the flooded condition maintained for analysis later. Fresh and dry weights, and moisture contents are presented in Table 11.

## 2 SPARTINA - GREENHOUSE

4 replications, of 6 different sediments (the 5 described in the previous report, plus a reference sediment from the Dyfi estuary), and the WES reference soil were treated with both lime and fertiliser. Three plants were transplanted into each pot on June 22nd. Initial growth was in tapwater, to ensure good establishment, and slow progress was made at first. However, all seedlings survived, and the tapwater was replaced by 15 p.p.t saltwater (prepared from WES Instant Ocean), after 3 weeks.

Good and rapid growth followed, and the saline water replaced fortnightly, the 15 p.p.t level maintained. Again, plants were kept submerged to 5 cm depth, and the buckets scrubbed periodically to restrain algal growth. Plants were also sprayed with freshwater periodically, to remove any surface accumulation of salt on the leaves.

## 2.2 SPARTINA - FIELD

Protective nylon mesh covers were kept in position all through the growing period, primarily to protect the plants from destruction by sheep. Slow growth was observed, but all plants survived, and appeared to be thriving well compared with the natural Spartina marsh. Photographs were taken at both high and low tide.

## 2.3 HARVEST - GREENHOUSE

The greenhouse plants grew vigorously. For 2 weeks prior to harvest, no surface spraying was performed, and the surface salt deposits allowed to accumulate. Immediately before harvest, these deposits were collected by wiping the surfaces of the leaves with a commercial "wet wipe" which was then frozen and stored for later analysis, with a view to tracing the movements of elements through the plant.

The plants were then harvested, all above ground parts collected, washed in the usual way, and fresh and dry weights recorded. These along with height data, and moisture content are presented in Table 12.



## 2.4 HARVEST - FIELD

The field experiment was harvested on November 16th, slightly later than planned, to allow the plants to reach maximum development. Although a 120 day harvest period had been agreed, it was felt that at this stage, the plants had not reached full maturity, and so to compensate for the slow initiation of the seedlings, a further 3 weeks growing time was allowed. The plants were harvested, washed and dried in the usual way. Fresh and dry weights were again recorded and are presented in Table 13 along with data on height and percentage water content.

The pots were left embedded in the Dyfi, at the experimental site, to allow a second year's growth and harvest, if necessary.

## 3 GENERAL INFORMATION AND RESPONSES TO LIMING

1) Photographs were taken of all plants at harvest.

2) Responses to liming on a fresh weight basis, of all species have been calculated and are presented in Appendix 1.

Generally positive responses were observed, particularly on the Parys Mountain and Frongoch soils. Little or no response was observed on the the Ystwyth soils and in the Halkyn soils, effects varied. Some started negatively, and became positive; others remained negative (radish), and others were positive all the time (wheat).

All differences between limed and unlimed pots decreased with time, as did the yield, probably a reflection on the effectiveness of the fertiliser treatment.

3) *Spartina* has been collected from all the estuaries for comparison with that grown in the same sediments in the Dyfi estuary.

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PROSPECTIVE

During the next year, a complete analysis of all vegetable material will be made, for the elements Pb, Zn, Cu, Cd and Hg. The soils will be stored as appropriate (i.e flooded conditions maintained), and analysed according to the schedule agreed with Dr Lee.

R+D-4129-REN

Heavy metal uptake by Cyperus esculentus, Spartina alterniflora and  
Agronomic crops from contaminated soils and sediments

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Fourth Interim Report

January 1983 through July 1983

CONTRACT NUMBER DAJA37 - 82 - C - 0195

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### Introduction

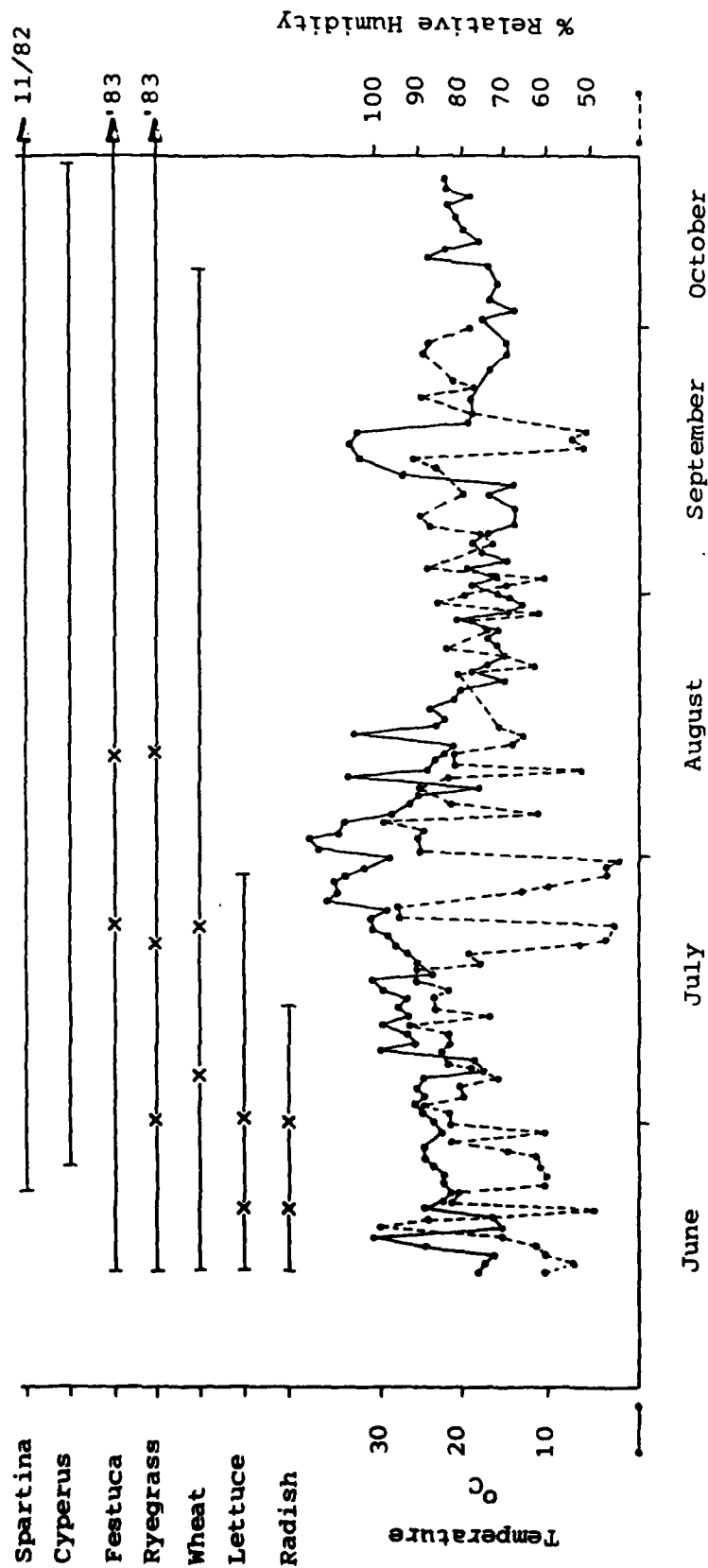
This report is concerned mainly with the analysis of soils and vegetation, which has been carried out during this particular research period, and the presentation of these preliminary results. Further details of the harvesting and plant yields are also included where they were not available for the previous report. Most of the analysis is now complete for both the freshwater experiments and the Spartina experiment, hence the next period of research will focus mainly on the statistical analysis of the data presented here.

### Performance of Agronomic crops and Cyperus esculentus

The third interim report, July 1982 - January 1983 described the growth, management and harvest of the agronomic crops, Cyperus esculentus and Spartina alterniflora. Details of the growth environment were closely monitored, particularly temperature and relative humidity, and are presented together with the management regime in Fig 1. These data are pertinent to the performance of all plants in the early stages, and wheat, Cyperus and Spartina throughout the experiments. From this diagram can be seen the variable nature of the British summer on a daily basis, and that the temperatures are far below those of WES.

It is worth noting that in the case of Cyperus, plants were harvested at maximum growth stage, much later than the 45 day period suggested by WES (at which stage the plants were nowhere near the maximum vegetative growth stage). Growth in the U.K was much slower than at WES, presumably due to reduced temperatures and a longer

**Figure 1** Temperature and relative humidity data for summer 1982, also indicating plant management regime (X denotes thin/cut)



comparable growth stage rather than growth period was considered more relevant.

#### Effects of Liming

As outlined in the third report, half of the soils were left in their natural state, while a repeat set of experiments was performed under limed conditions - lime was added at a rate sufficient to bring the soils close to pH 7. The Somerset soil, already at pH 7 was not repeated.

All of the plants fared better in the limed compared with the unlimed soils in every case except that of Halkyn Mountain, where a strange, reversed growth pattern was observed. When actual yields are compared, the effects of liming can be seen clearly (Table 1). Increased yields occurred in most plants and the greater the amount of lime added, the more is the response - Parys Mountain particularly, with a normal pH of 5.9 showed marked yield increases for all crops.

Table 1

EFFECTS OF LIMING ON PLANT YIELD

Site	Lettuce		Lolium (Cut 2)		Festuca (Cut 2)	
	UL	L	DL	L	UL	L
Ystwyth	148 ± 47	72 ± 28.9	43.6 ± 11.2	42.3 ± 12.3	5.2 ± 1.1	7.2 ± 2.4
Frongoch	20 ± 4.2	64 ± 18.4	16.3 ± 5.9	52.9 ± 9.6	4.2 ± 2.0	4.7 ± 0.8
Parys Mt.	110 ± 23.3	168 ± 32.1	14.3 ± 7.7	65.2 ± 4.2	4.5 ± 2.1	30.3 ± 5.8
Halkyn Mt.	95 ± 28.8	66 ± 7.6	32.7 ± 2.7	35.5 ± 4.3	6.6 ± 1.7	5.8 ± 4.3
Somerset	257 ± 65.7		66.5 ± 30.4		19.1 ± 4.6	

Plant yield contd

Site	Wheat Wt		Wheat Ht		Wheat HT		Radish Leaf		Radish Bulb	
	UL	L	UL	L	UL	L	UL	L	UL	L
Ystwyth	12.7 ± 1.8	11.9 ± 0.7	68 ± 1.5	67 ± 3	90.8 ± 25.8	49.6 ± 9.7	172.7 ± 31.8	104.0 ± 11.0		
Frongoch	10.1 ± 0.2	18.1 ± 2.7	59 ± 6.2	75 ± 5.1	24.7 ± 7.2	39.0 ± 4.6	30.8 ± 12.6	76.8 ± 12.2		
Parys Mt.	7.6 ± 1.8	21.2 ± 9.9	28 ± 0	75 ± 8.5	39.0 ± 4.6	29.7 ± 7.9	47.2 ± 21.7	206.0 ± 71		
Haikyn Mt.	5.5 ± 1.5	11.7 ± 1.5	43 ± 7	57 ± 8.7	67.2 ± 7.8	29.7 ± 7.1	127.0 ± 31.5	65.3 ± 26.6		
Somerset	13.2 ± 4.2		59 ± 3		101 ± 20.3		214.0 ± 34.3			



### Soils Analysis

The selection of the five sites for this set of experiments was based on the initial sampling and analysis described in the second interim report. A map of the locations is given here for reference. (Fig 2).

However, during the experimental stage, a more detailed sampling regime was performed on each soil for the different treatments (L and UL) and also to compare the flooded and upland conditions. Exact growth conditions were maintained during the sampling i.e. flooded soils were kept flooded at all times.

Profile samples were taken from each pot and bulked according to treatment. Soils were air dried, ground and passed through a nylon sieve of 2 mm aperture (see Appendix 1 for details). The following analyses were then performed.

#### For all Soils;

##### 1) Soil reaction

The pH of each soil under both limed and unlimed conditions was determined by a pH meter after equilibration with distilled water for 30 minutes, according to the method outlined in Appendix 1. The results are presented in Table 2. From this determination was calculated the lime requirement on a per pot basis. A second pH determination was made after this had been added and both the lime requirement and the pH after lime addition are given in Table 2. In all cases the addition of lime raised the pH, to about 7 except Parys Mt. where the resultant pH was slightly lower at 6.4.

Figure 2

DISTRIBUTION OF SOIL SAMPLING SITES

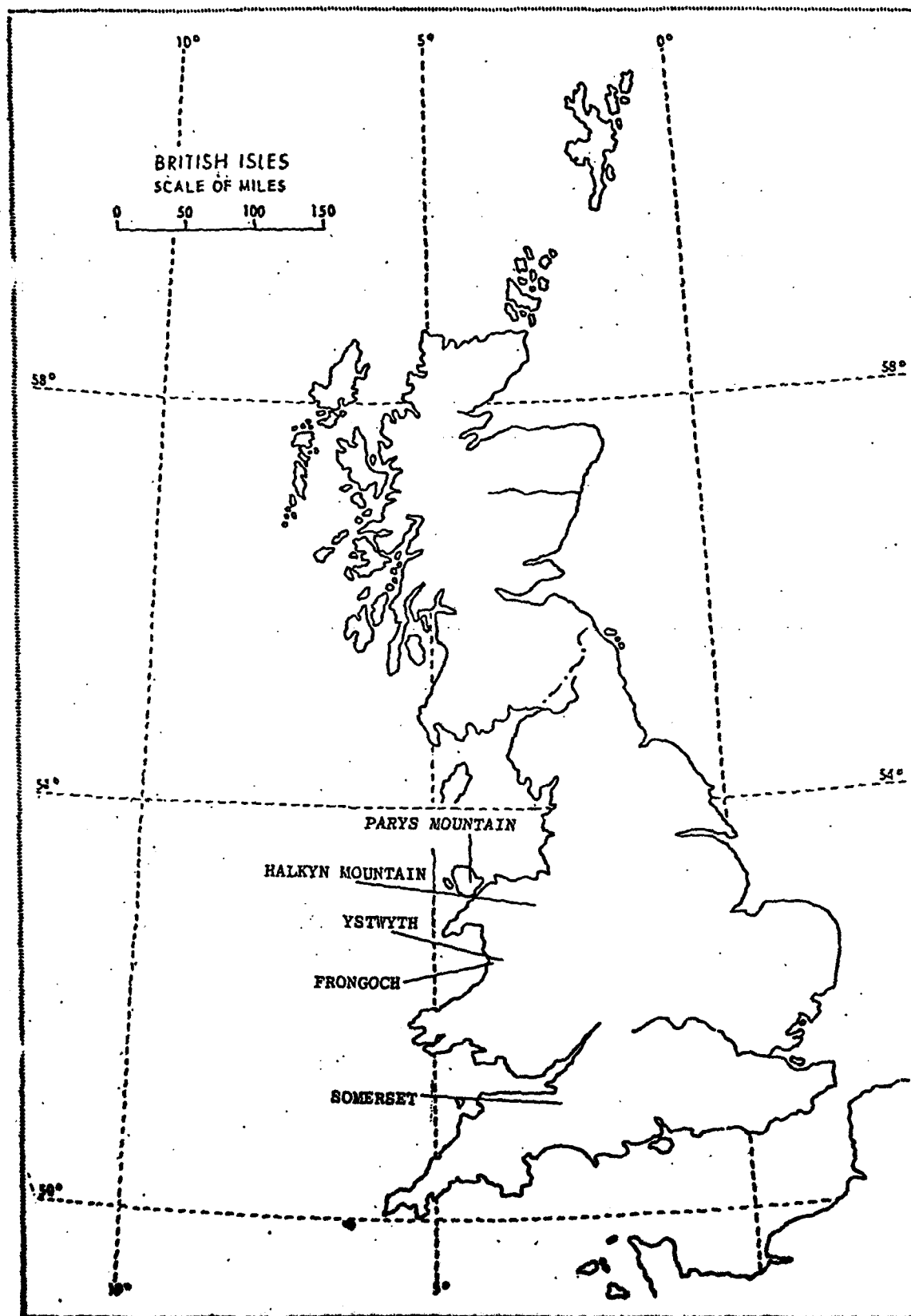


Table 2

% organic matter, pH and lime requirements of soils

Site	Treatment	% OM	pH	Lime Req#	Final pH
Ystwyth	UL	6.3	5.7	66	-
	L	6.7	-	-	7.1
Frongoch	UL	9.4	6.6	10	-
	L	11.8	-	-	7.1
Parys Mt.	UL	12.9	5.9	15	-
	L	11.9	-	-	6.4
Halkyn Mt.	UL	15.0	5.75	60	-
	L	13.4	-	-	7.4
Somerset	UL	10.0	7.2	-	-

# cwt/acre

## 2) Soil organic matter content

Soil organic matter was determined gravimetrically following loss on ignition at 430 °C for 24 hours according to the method outlined in Appendix 1. The results are presented in Table 2 together with the pH and lime requirements of soils.

## 3) Cation Exchange Capacity

Major exchangeable cations of Ca, Mg, Na and K were determined using flame AAS following extraction into NH<sub>4</sub> OAc (pH 7) according to Horn et al (1982). Exchangeable hydrogen was determined following the method of Peech (1965). CEC was derived from the addition of exchangeable hydrogen to the exchangeable cations and is presented for each soil in Table 3.

## 4) Particle size distribution

The percentage by weight of each of the size classes used by the USDA to denote sand, silt and clay fractions in soil was derived by the Bouyoucos Hydrometer method described in Appendix 1 for the silt and clay fractions. Sand fractions were derived by sieving at the appropriate aperture. The results are presented in Table 4.

## 5) Total Metal Contents

The elements Pb, Zn, Cu, Cd, Ni, Fe, Mn, Co, Cr, Al, were determined by AAS on the five soils under both limed and unlimed conditions following digestion with HNO<sub>3</sub> by the method outlined in Appendix 1. The results for all elements are presented in Table 5. From this table it can be seen that the original intention of having a soil high in at least one of the four major heavy metals under consideration, has been followed except in the case of zinc. It was hoped that the Halkyn Mountain soil would prove high in zinc but

Table 3

## CATION EXCHANGE CAPACITY OF UPLAND SOILS (meq/100g)

Site	Trt	Me	Ca	K	Na
Ystwyth	UL	0.18 ± 0.00	2.36 ± 0.02	0.04 ± 0.00	0.46 ± 0.00
	L	0.16 ± 0.00	6.88 ± 0.21	0.04 ± 0.00	0.54 ± 0.02
Frongoch	UL	0.72 ± 0.06	2.81 ± 0.18	0.15 ± 0.01	0.18 ± 0.00
	L	0.58 ± 0.04	4.27 ± 0.09	0.08 ± 0.00	0.40 ± 0.00
Parys Mt.	UL	0.18 ± 0.05	0.55 ± 0.01	0.13 ± 0.00	0.28 ± 0.02
	L	0.22 ± 0.02	2.08 ± 0.17	0.19 ± 0.00	0.48 ± 0.02
Halkyn Mt.	UL	0.24 ± 0.00	5.91 ± 0.04	0.19 ± 0.00	0.35 ± 0.00
	L	0.19 ± 0.00	1.10 ± 0.01	0.15 ± 0.00	0.41 ± 0.00
Somerzet	UL	1.2 ± 0.19	5.70 ± 0.02	0.20 ± 0.00	0.21 ± 0.00

TABLE 4

PARTICLE SIZE DISTRIBUTION OF UPLAND SOILS U.S.D.A. LIMITS

Site	Trt	2000 - 200 Coarse Sand	200 - 50 Fine Sand	50 - 2 Silt	< 2 Clay
Ystwyth	UL	51.2	24.1	20.0	5.75
	L	48.5	23.3	23.0	5.25
Frongoch	UL	48.4	22.4	21.0	8.25
	L	50.2	28.5	26.0	5.25
Parys Mt.	UL	49.2	24.1	22.0	4.75
	L	47.6	23.2	23.0	4.95
Halkyn Mt.	UL	66.8	11.8	13.0	1.25
	L	69.0	15.3	14.0	1.75
Somerset	UL	50.8	22.5	26.0	0.75

Table 5

TOTAL + DTPA EXTRACTABLE METAL LEVELS IN UPLAND SOILS

Site	Trt	Hg/Kg		Zn		Cu		Cd	
		Pb		TOTAL	DTPA	TOTAL	DTPA	TOTAL	DTPA
		TOTAL	DTPA						
Ystwyth	UL	488 ± 16	187 ± 36.5	256 ± 11.6	12.4 ± 2.7	12.2 ± 0.4	4.6 ± 0.8	0.42 ± 0.01	0.4
	L	466 ± 13	174 ± 23.0	263 ± 13.4	6.9 ± 0.1	12.7 ± 0.1	4.2 ± 0.3	0.43 ± 0.00	0.2
Frongoch	UL	23 ± 0.7	7.8 ± 0.0	49.1 ± 3.8	1.25 ± 0	6.6 ± 0	2.4 ± 0.1	0.30 ± 0.01	0.2
	L	23 ± 1.3	6.9 ± 0.2	48.4 ± 4.15	0.75 ± 0.1	7.1 ± 0.1	2.1 ± 0	0.21 ± 0.03	0.1
Parys Mt.	UL	597 ± 54	204 ± 0.7	114 ± 11.3	2.3 ± 0.2	468 ± 48	180 ± 18	0.30 ± 0.01	0.2
	L	586 ± 7.5	185 ± 2.8	111 ± 7.5	2.1 ± 0.0	417 ± 6.2	124 ± 4	0.23 ± 0.02	0.1
Halkyn Mt.	UL	419 ± 1.8	20.1 ± 0.9	762 ± 132	53.5 ± 2.7	196 ± 24	14.2 ± 0.3	0.78 ± 0.05	0.4
	L	324 ± 27.0	30 ± 1.3	835 ± 79	49.5 ± 3.8	151 ± 5.9	12.8 ± 0	0.74 ± 0.03	0.1
Somerset	UL	445 ± 7.0	48.9 ± 0.4	3408 ± 81.3	407 ± 1	6.7 ± 0.4	2.8 ± 0	109 ± 4.5	27.1

mg/kg									
Cu		Cd		Mn		Fe		Cr	
TOTAL	DTPA	TOTAL	DTPA	TOTAL	DTPA	TOTAL	DTPA	TOTAL	DTPA
12.2 ± 0.4	4.6 ± 0.4	0.42 ± 0.01	0.4 ± 0	617 ± 108	14.2 ± 3.5	24,185 ± 98	52.8 ± 0.4	28.9 ± 0.7	0.11 ± 0.1
12.7 ± 0.1	4.3 ± 0.3	0.43 ± 0.00	0.23 ± 0.02	635 ± 8.9	8.5 ± 0.07	23,733 ± 1,266	40.1 ± 0.7	19.8 ± 1.5	0.31 ± 0.1
6.6 ± 0	3.4 ± 0.1	0.30 ± 0.01	0.25 ± 0	250 ± 4.8	8.1 ± 0.05	17,779 ± 442	50.8 ± 1.9	21.1 ± 0.2	0.14 ± 0.0
7.1 ± 0.1	3.1 ± 0	0.21 ± 0.03	0.15 ± 0.1	339 ± 10.8	7.1 ± 0.07	14,995 ± 1,385	36.7 ± 3.9	16.4 ± 0.9	0.01 ± 0.0
488 ± 48	150 ± 18	0.30 ± 0.01	0.25 ± 0	248 ± 1.6	9.3 ± 0.17	17,981 ± 1,846	198 ± 3.5	11.1 ± 2.7	0.08 ± 0.0
417 ± 6.2	134 ± 4	0.23 ± 0.02	0.18 ± 0.02	289 ± 1.1	7.2 ± 0.08	17,556 ± 753	229 ± 3.5	13.9 ± 1.5	0.08 ± 0.0
198 ± 24	14.3 ± 0.3	0.78 ± 0.05	0.4 ± 0.05	901 ± 1.4	5.7 ± 0.1	12,232 ± 136	11.8 ± 2.2	21.7 ± 2.7	0.12 ± 0.0
151 ± 5.9	12.8 ± 0	0.74 ± 0.03	0.18 ± 0	627 ± 45.3	5.4 ± 0	15,792 ± 391	15.2 ± 0.8	27.1 ± 0.0	0.12 ± 0.0
8.7 ± 0.4	3.9 ± 0	109 ± 4.5	27.5 ± 0.5	1593 ± 40.7	19.8 ± 0.2	14,903 ± 986	23.6 ± 0.7	3.45 ± 0.1	0.01 ± 0.0



are all present.

6) Easily exchangeable - DTPA extractable metal contents of soils

i) Upland soils- Soils were extracted by shaking air dried soil with DTPA (prepared according to the method of Lindsay and Norvell 1978) at a ratio of 1:5, for 2 hours. The extracts were then filtered into a conical flask through a Whatman 542 filter paper. The same set of elements as for the total extraction were determined by flame AAS, and are presented together with the total values in Table 5.

ii) Flooded soils. At no time were the soils allowed to dry out (oxidise) and hence alter the availability of the elements to be tested. Flooded soils were extracted with DTPA at a ratio of 5:1 by shaking for 2 hours. Correct proportions of DTPA : Soil were ensured by calculating the % moisture in the flooded soil by drying a replicate sample. The dry weight of soil in the flooded sample and therefore the correct amount of DTPA to be added can then be calculated. The extracts were filtered through a Whatman 542 filter paper into a conical flask and all elements determined by flame AAS, and are presented in Table 6.

\*

- N.B. The soil moisture must be taken into account when adding DTPA. In order to maintain the correct DTPA strength and avoid dilution by the soil moisture, distilled water was added such that, together with the existing soil water (calculated from loss in weight when dried), it comprised half the required amount of DTPA. Double strength DTPA was then added to the required amount.

The ratios of total : extractable levels of metals were

Table 6

DTPA EXTRACTABLE HEAVY METAL LEVELS - FLOODED SOILS

	Pb	Zn	Cu	Cd	Fe	Mn	Co	Ni
Ystwyth	UL 46.6	16.9	7.8	0.36	465	395	2.55	0.15
	L 46.2	14.9	5.7	0.18	505	195	0.65	0.25
Frongoch	UL 2.66	2.63	6	0.05	1050	242	0.55	0.05
	L 2.71	0.81	6.2	0.05	940	228	0.75	0.05
Parys Mt.	UL 444	7.03	298	0.12	880	84	3.15	0.65
	L 221	4.03	120	0.09	1090	87	2.05	0.20
Halkyn Mt.	UL 35.4	33.8	13.8	0.10	21.1	2.95	0.15	0.00
	L 27.4	33.4	14.7	0.12	23.8	2.88	0.15	0.05
Somerset	UL 87.5	332	2.7	6.75	28.3	730	3.05	0.70

calculated for all elements and are presented for both flooded and upland conditions in Tables 7 and 8. Reference to these figures should give some indication as to the availability of the elements under oxidised and reduced conditions, and some differences can be observed. DTPA extracts generally more metals from upland than reduced soils.

Table 7

RATIO OF TOTAL : DTPA EXTRACTABLE HEAVY METAL LEVELS - UPLAND SOILS

	Pb	Zn	Cu	Cd	Mn	Fe	Cr	
Ystwyth	UL	0.38	0.05	0.38	0.95	0.02	0.002	0.004
	L	0.37	0.03	0.33	0.53	0.01	0.002	0.012
Frongoch	UL	0.34	0.03	0.36	0.83	0.03	0.003	0.007
	L	0.30	0.02	0.30	0.71	0.02	0.002	0.001
Parys Mt.	UL	0.34	0.02	0.31	0.83	0.04	0.011	0.007
	L	0.32	0.02	0.32	0.78	0.03	0.013	0.006
Halkyn Mt.	UL	0.05	0.07	0.07	0.51	0.01	0.001	0.005
	L	0.09	0.06	0.08	0.24	0.01	0.001	0.004
Somerset	UL	0.11	0.12	0.32	0.25	0.01	0.002	0.003

Table 8

RATIO OF TOTAL : DTPA EXTRACTABLE HEAVY METAL LEVELS - FLOODED SOILS

		Pb	Zn	Cu	Cd	Mn	Fe	Cr
Ystwyth	UL	0.09	0.001	0.63	0.85	0.64	0.019	
	L	0.09	0.001	0.44	0.42	0.31	0.021	
Frongoch	UL	0.11	0.053	0.91	0.16	0.96	0.059	
	L	0.12	0.016	0.87	0.23	0.67	0.062	
Parys Mt.	UL	0.74	0.062	0.61	0.4	0.34	0.048	
	L	0.37	0.036	0.29	0.39	0.30	0.062	
Halkyn Mt.	UL	0.08	0.044	0.07	0.13	0.003	0.002	
	L	0.08	0.040	0.09	0.16	0.004	0.002	
Somerset	UL	0.19	0.097	0.31	0.06	0.45	0.002	

### Analysis of vegetation

Following drying, the vegetation was finely chopped using stainless steel scissors and disaggregated using a pestle and mortar. % ash content of each sample was derived from ignition of the sample at 550 °C according to the method outlined in Appendix 1. Ash material was then digested with HNO<sub>3</sub> according to the method in Appendix 1, and the extract characterised for Pb, Zn, Cu and Cd either by flame (Zn, Cu) or flameless AAS (Pb, Cd). 20% replication was maintained and the sample repeated if the co-efficient of variation between replicates was above 10%. Accuracy was checked by the routine analysis of NBS reference material, in this case, Tomato and Spinach leaves. % Average recoveries were for tomato and spinach were as follows :

	Pb	Zn	Cu
Spinach	111	98	91
Tomato	106	94	91

## Results

### Agronomic crops

Results were calculated in terms of mg/kg on a dry weight basis and are presented for the agronomic crops radish, lettuce and grass in Tables 9, 10, 11 & 12.

In the case of radish, both leaves and bulbs were analysed, and from Table 9 it can be seen that uptake patterns were similar, although in some cases, the bulbs accumulated more than the leaves e.g. Parys Mt. Pb. Liming generally decreased uptake, particularly of lead from most of the soils, and also zinc. Reversed results are seen in the Halkyn Mountain experiment, which follows the strange growth responses observed - no explanation can be offered at the moment and the experiment is being repeated to check the results.

For lettuce (Table 10), similar patterns are observed although some reversal of liming effect is noted in the Ystwyth soil. No elevation of copper was found in the Parys Mt. soil as might have been expected, but high Cd levels are observed in the Somerset soil.

For Lolium and Festuca, the second cut of four was analysed during this research period, and the effects of liming on particular Pb levels can be seen clearly from Tables 11 and 12. Reduced uptake was observed in all cases with liming, except for Halkyn Mountain which is proving somewhat anomalous. Uptake patterns generally reflect the contents of the underlying soils.

### Wheat

The results of the wheat experiment are presented in Tables 13 and 14. Both wheat heads and stalks were analysed. Again, underlying soil levels are reflected for all the metals, and liming

Table 9

Heavy metal contents of Radish leaves grown on contaminated soils ( $\bar{x} \pm S.D$ )

Site	Trt	mg/kg			
		Pb	Zn	Cu	Cd
Ystwyth	UL	8.40 $\pm$ 0.57	116.7 $\pm$ 11.3	3.35 $\pm$ 0.16	1.16 $\pm$ 0.19
	L	6.80 $\pm$ 0.83	40.6 $\pm$ 5.3	2.75 $\pm$ 0.18	0.74 $\pm$ 0.08
Frongoch	UL	3.02 $\pm$ 0.22	36.9 $\pm$ 8.30	2.91 $\pm$ 0.28	0.46 $\pm$ 0.08
	L	2.86 $\pm$ 0.45	29.5 $\pm$ 3.40	3.38 $\pm$ 0.53	0.35 $\pm$ 0.06
Parys Mt.	UL	9.27 $\pm$ 2.04	137.5 $\pm$ 28.4	48.90 $\pm$ 6.9	1.49 $\pm$ 0.43
	L	5.68 $\pm$ 0.46	44.7 $\pm$ 4.7	15.7 $\pm$ 3.1	0.34 $\pm$ 0.04
Halkyn Mt.	UL	2.69 $\pm$ 0.21	129.4 $\pm$ 24.7	4.37 $\pm$ 0.77	0.22 $\pm$ 0.03
	L	3.19 $\pm$ 0.31	128.4 $\pm$ 20.7	5.81 $\pm$ 0.64	0.36 $\pm$ 0.05
Somerset	UL	3.86 $\pm$ 1.90	253.7 $\pm$ 20.2	6.34 $\pm$ 1.20	16.40 $\pm$ 0.50

Heavy metal contents of Radish bulbs grown on contaminated soils ( $\bar{x} \pm S.D$ )

Site	Trt	mg/kg			
		Pb	Zn	Cu	Cd
Ystwyth	UL	4.79 $\pm$ 0.9	106.9 $\pm$ 7.0	3.21 $\pm$ 0.36	1.85 $\pm$ 0.03
	L	4.52 $\pm$ 0.64	59.6 $\pm$ 19	2.37 $\pm$ 0.54	0.14 $\pm$ 0.02
Frongoch	UL	1.96 $\pm$ 0.6	43.2 $\pm$ 7.6	2.23 $\pm$ 0.32	0.31 $\pm$ 0.02
	L	1.05 $\pm$ 0.05	34.7 $\pm$ 2.45	2.13 $\pm$ 0.36	0.26 $\pm$ 0.09
Parys Mt.	UL	27.4 $\pm$ 3.7	125.8 $\pm$ 13.5	35.54 $\pm$ 6.64	0.56 $\pm$ 0.05
	L	6.31 $\pm$ 1.2	51.3 $\pm$ 9.8	13.24 $\pm$ 1.8	0.26 $\pm$ 0
Halkyn MT.	UL	0.33 $\pm$ 0.05	90.0 $\pm$ 17.4	5.29 $\pm$ 0.56	0.20 $\pm$ 0.03
	L	0.49 $\pm$ 0.05	102.1 $\pm$ 19.4	4.89 $\pm$ 0.60	0.18 $\pm$ 0.02
Somerset	UL	2.23 $\pm$ 0.25	148.5 $\pm$ 30.7	27.00 $\pm$ 6.64	11.5 $\pm$ 0.58



Table 10  
Heavy metal contents of Lettuce grown on contaminated soils ( $\bar{x}$  + S.D)

Site	Trt	mg/kg			
		Pb	Zn	Cu	Cd
Ystwyth	UL	3.30 ± 0.8	98.3 ± 30.7	4.5 ± 1.7	1.17 ± 0.31
	L	5.67 ± 3.4	23.6 ± 3.1	3.0 ± 0.45	0.27 ± 0.02
Frongoch	UL	1.51 ± 0.18	15.2 ± 4.1	3.6 ± 0.76	0.17 ± 0
	L	1.23 ± 0.27	10.8 ± 1.6	2.5 ± 0.33	0.16 ± 0.05
Parys Mt.	UL	1.91 ± 0.39	35.7 ± 5.4	6.2 ± 0.42	0.36 ± 0.05
	L	4.05 ± 1.9	45.8 ± 9.2	9.8 ± 2.18	0.41 ± 0.14
Halkyn Mt.	UL	0.75 ± 0.08	30.4 ± 3.5	3.9 ± 0.25	0.18 ± 0
	L	1.48 ± 0.35	42.5 ± 8.1	4.9 ± 1.26	0.63 ± 0.29
Somerset	UL	1.02 ± 0.17	155.8 ± 36.2	4.9 ± 1.9	7.77 ± 0.04

Table 11  
Heavy metal contents of *Festuca rubra* grown on contaminated soils ( $\bar{x} \pm S.D$ )

Site	Trt	mg/kg			
		Pb	Zn	Cu	Cd
Ystwyth	UL	2.05 $\pm$ 0.56	24.03 $\pm$ 3.02	6.58 $\pm$ 0.42	0.28 $\pm$ 0.06
	L	1.55 $\pm$ 0.16	19.28 $\pm$ 4.5	4.20 $\pm$ 0.25	0.24 $\pm$ 0.02
Frongoch	UL	2.03 $\pm$ 0.58	22.4 $\pm$ 4.8	3.42 $\pm$ 0.45	0.23 $\pm$ 0.04
	L	2.33 $\pm$ 0.75	24.0 $\pm$ 10.4	3.94 $\pm$ 0.86	0.23 $\pm$ 0.01
Parys Mt.	UL	9.39 $\pm$ 0.58	47.5 $\pm$ 1.75	13.0 $\pm$ 1.22	1.02 $\pm$ 0.15
	L	3.32 $\pm$ 0.69	32.1 $\pm$ 4.2	9.12 $\pm$ 1.31	0.25 $\pm$ 0.03
Halkyn Mt.	UL	2.20 $\pm$ 0.84	26.9 $\pm$ 2.49	6.42 $\pm$ 0.74	0.13 $\pm$ 0.03
	L	7.86 $\pm$ 0.03	39.0 $\pm$ 6.2	9.96 $\pm$ 2.73	0.35 $\pm$ 0.05
Somerset	UL	1.23 $\pm$ 0.24	31.2 $\pm$ 11	4.75 $\pm$ 1.75	1.46 $\pm$ 0.26

Table 12

HEAVY METAL CONTENTS OF *LOLIUM MULTIFLORUM* GROWN ON CONTAMINATED SOILS ( $\bar{x} \pm \text{S.D.}$ )

Site	Trt	mg/kg			
		Pb	Zn	Cu	Cd
Ystwyth	UL	3.73 $\pm$ 0.76	50.5 $\pm$ 13.7	5.73 $\pm$ 0.29	0.43 $\pm$ 0.05
	L	2.86 $\pm$ 1.01	35.2 $\pm$ 9.2	5.27 $\pm$ 1.33	0.38 $\pm$ 0.16
Frongoch	UL	1.91 $\pm$ 0.53	11.3 $\pm$ 1.6	3.07 $\pm$ 1.12	0.25 $\pm$ 0.09
	L	1.53 $\pm$ 0.16	13.1 $\pm$ 3.0	4.19 $\pm$ 1.08	0.13 $\pm$ 0.02
Parys Mt.	UL	7.38 $\pm$ 1.37	33.1 $\pm$ 7.7	20.4 $\pm$ 0.52	0.34 $\pm$ 0.2
	L	3.63 $\pm$ 0.78	38.5 $\pm$ 4.02	15.3 $\pm$ 3.5	0.13 $\pm$ 0.03
Halkyn Mt.	UL	2.69 $\pm$ 0.55	70.3 $\pm$ 13.3	9.95 $\pm$ 1.3	0.16 $\pm$ 0.03
	L	2.66 $\pm$ 0.24	69.7 $\pm$ 12.1	7.4 $\pm$ 0.2	0.34 $\pm$ 0.66
Somerset	UL	2.24 $\pm$ 0.78	25.0 $\pm$ 1.45	5.11 $\pm$ 0.48	1.41 $\pm$ 0.31

H

Table 13

HEAVY METAL CONTENTS OF WHEAT STALKS GROWN ON CONTAMINATED SOILS ( $\bar{x} \pm \text{S.D.}$ )

Site	Trt	Mg/Kg				Cu	Cd
		Pb	Zn				
Yatwyth	UL	3.75 $\pm$ 0.8	29.2 $\pm$ 7.8		1.42 $\pm$ 0.2		0.6 $\pm$ 0.22
	L	2.82 $\pm$ 0.7	13.6 $\pm$ 3.9		1.47 $\pm$ 0.2		0.34 $\pm$ 0.23
Frongoch	UL	2.13 $\pm$ 0.8	15.3 $\pm$ 3.6		1.91 $\pm$ 0.8		0.13 $\pm$ 0.08
	L	1.97 $\pm$ 0.2	10.2 $\pm$ 5.3		1.11 $\pm$ 0.4		0.11 $\pm$ 0.01
Parys Mt.	UL	14.9 $\pm$ 3.7	103 $\pm$ 9.9		32.2 $\pm$ 6.1		0.98 $\pm$ 0.16
	L	6.8 $\pm$ 1.9	20 $\pm$ 6.3		1.64 $\pm$ 0.3		0.21 $\pm$ 0.00
Halkyn Mt.	UL	3.54 $\pm$ 1.3	48.0 $\pm$ 4.5		0.93 $\pm$ 0.41		0.07 $\pm$ 0.00
	L	2.31 $\pm$ 0.7	24.4 $\pm$ 4.7		0.86 $\pm$ 0.3		0.06 $\pm$ 0.00
Somerset	UL	3.18 $\pm$ 0.8	101 $\pm$ 23.8		1.17 $\pm$ 0.9		1.21 $\pm$ 0.25

Table 14

HEAVY METAL CONTENTS OF WHEAT HEADS GROWN ON CONTAMINATED SOILS ( $\bar{x} \pm S.D.$ )

Site	Trt	Mg/Kg			
		Pb	Zn	Cu	Cd
Yatwyth	UL	3.84 $\pm$ 0.37	31.5 $\pm$ 10.4	3.57 $\pm$ 0.28	0.66 $\pm$ 0.29
	L	2.54 $\pm$ 0.28	25.0 $\pm$ 13	3.15 $\pm$ 0.15	0.36 $\pm$ 0.08
Frongoch	UL	3.82 $\pm$ 1.01	19.3 $\pm$ 0.92	2.98 $\pm$ 0.06	0.31 $\pm$ 0.08
	L	1.95 $\pm$ 0.78	18.6 $\pm$ 3.0	3.16 $\pm$ 0.6	0.27 $\pm$ 0.16
Parys Mt.	UL	9.4 $\pm$ 5.9	92.9 $\pm$ 29	8.92 $\pm$ 1.6	0.89 $\pm$ 0.25
	L	3.54 $\pm$ 1.2	15.9 $\pm$ 3.24	3.58 $\pm$ 1.17	0.14 $\pm$ 0.1
Halkyn Mt.	UL	5.34 $\pm$ 3.0	29.5 $\pm$ 3.96	2.62 $\pm$ 0.3	0.14 $\pm$ 0.02
	L	1.66 $\pm$ 0.52	21.2 $\pm$ 1.95	2.33 $\pm$ 0.16	0.08 $\pm$ 0.03
Somerset	UL	2.68 $\pm$ 1.26	53.2 $\pm$ 8.1	3.54 $\pm$ 1.17	0.89 $\pm$ 0.18

has significantly reduced uptake of Pb and Zn particularly in all cases. Although wheat does not seem to concentrate heavy metal generally in the ripened heads, concentrations are higher in the heads for Cu in particular.

#### Cyperus esculentus

Eh probes received from WES were placed into the pots of both upland and flooded Cyperus experiments and readings taken every other day for upland soils and weekly for flooded soils. The results are presented in Figs 3 and 4 and from Fig 4 can be seen that 3 sediments were reduced immediately by flooding, but the Somerset and Halkyn sediments remained unchanged. From Fig 3, all the upland soils recorded highly oxidised conditions.

When plotted against pH, the Eh diagram become clearer (see Fig 5), and the two soils which did not register as reduced can be seen in the middle of the diagram between the two otherwise distinct groups of flooded and upland soils.

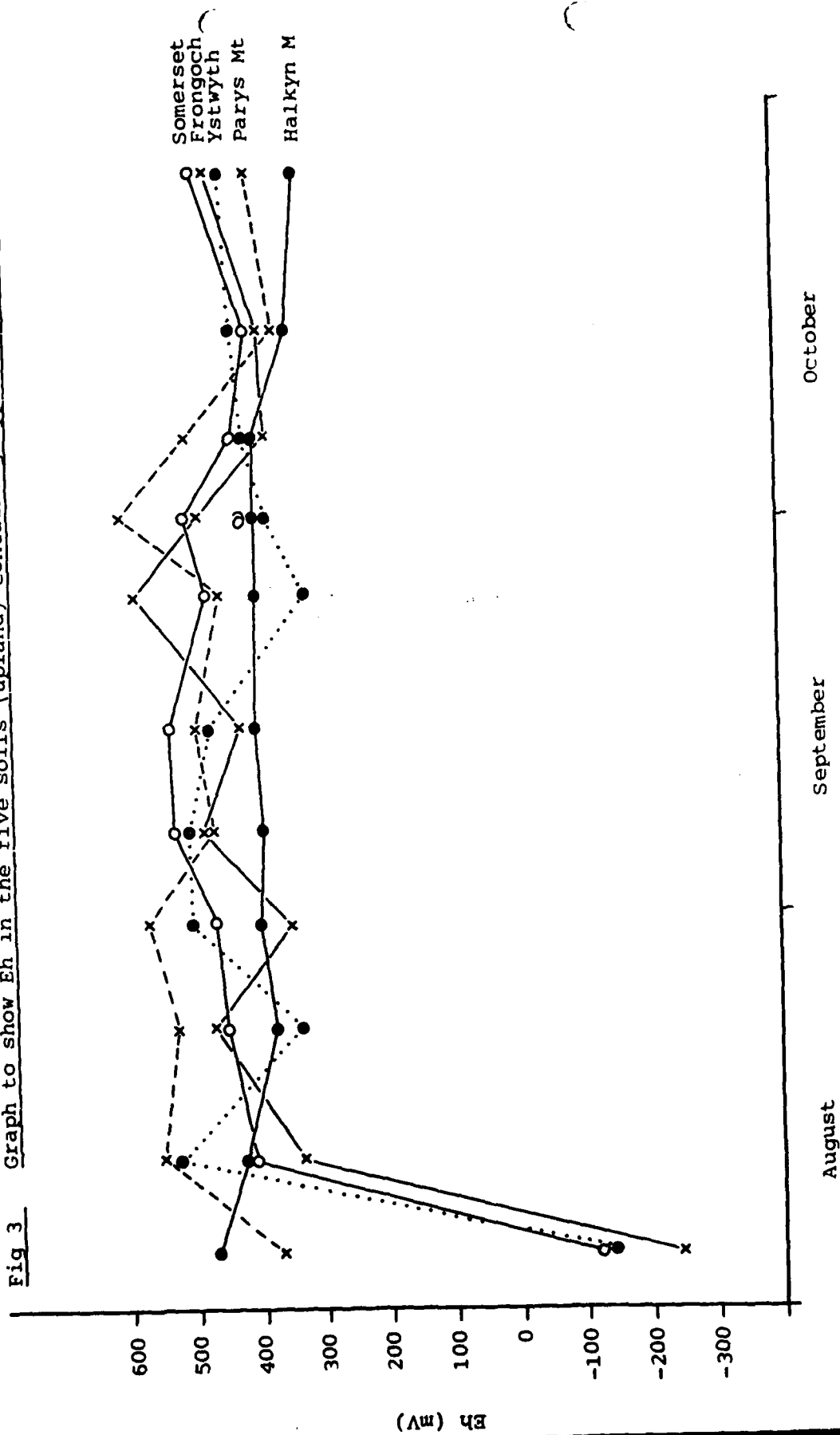
Plant yields were noted at harvest and differences can be observed between the flooded and upland experiments (see Table 15). Generally the flooded pots faired better, although this is probably a response to larger pots. Liming also improved growth, especially in the Parys Mt. soil in both flooded and upland conditions. All soils, however showed a liming response.

#### Results of analyses

Cyperus esculentus was analysed by the same method as described earlier for the agronomic crops. Results for flooded and upland tops are presented in Tables 16 and 17.

Patterns of uptake generally reflect soil distribution in

Fig 3 Graph to show Eh in the five soils (upland) containing *Cyperus esculentus*



**Fig 4** **Graph to show variations in Eh in flooded Cyperus esculentus experiment**

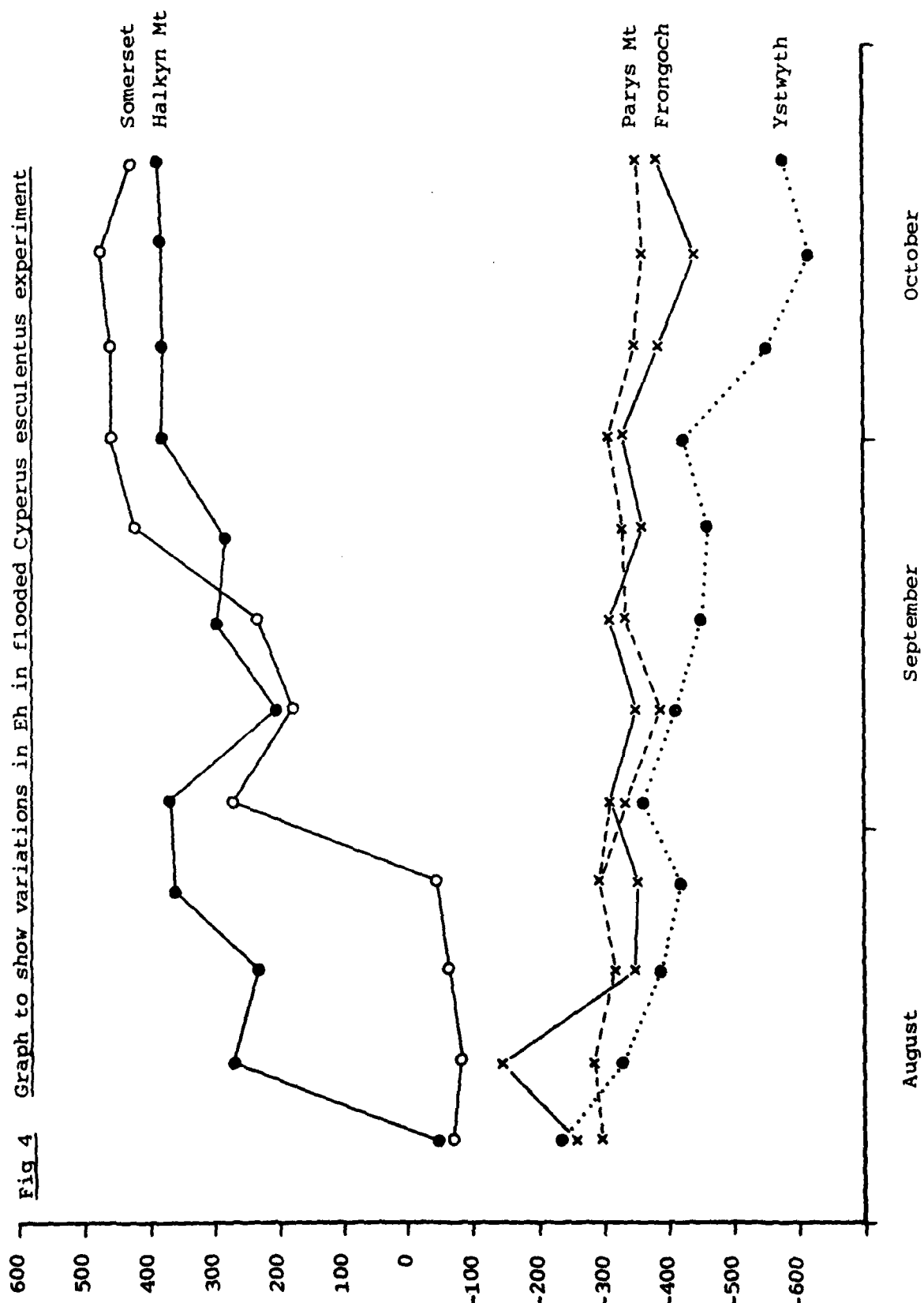




Figure 5

pH/Eh diagram for flooded (O) and upland soils

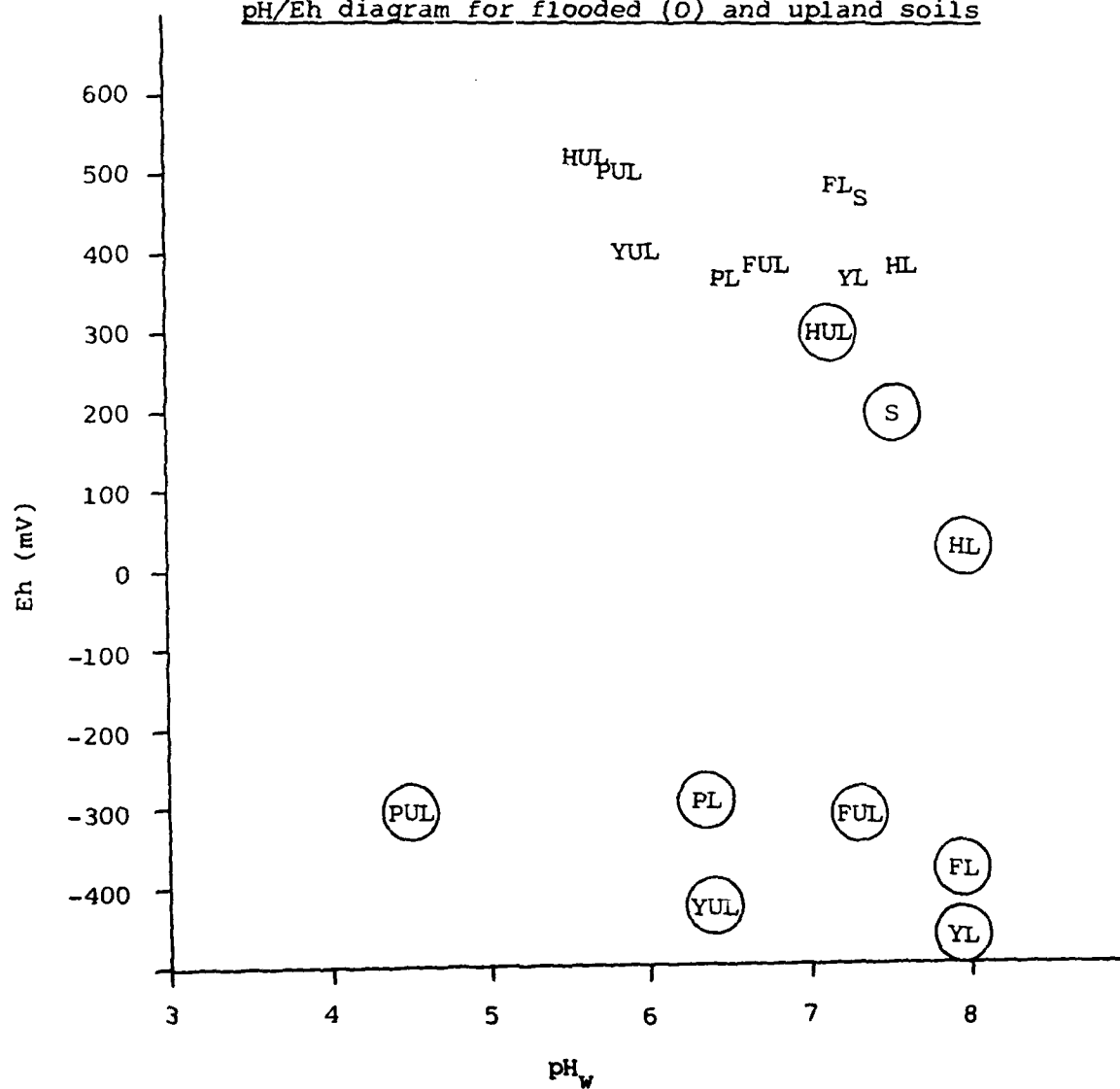


Table 15

## CYPERUS ESCULENTUS YIELD UNDER UPLAND AND FLOODED CONDITIONS

	Upland			Flooded					
				Leaves			Roots		
	UL	L		UL	L		UL	L	
Yst	48.1 ± 5.0	36.5 ± 23.2	69.4 ± 44.0	102.7 ± 67.1	94.5 ± 24.7	108.6 ± 14.8	81.1 ± 30.6	102.8 ± 14.3	
Fro	31.6 ± 6.0	43.5 ± 6.7	82.9 ± 14.5	83.7 ± 21.0	31.3 ± 4.5	37.5 ± 19.4	35.8 ± 14.3	49.6 ± 40.7	
P.M	3.3 ± 1.3	43.8 ± 28.2	21.6 ± 15.4	122.9 ± 67.8	22.4 ± 12.9	226.0 ± 53.9	11.0 ± 4.8	169.0 ± 41.7	
H.M	22.9 ± 5.2	32.7 ± 3.9	68.5 ± 36.1	83.2 ± 30.3	5.4 ± 0.8	42.5 ± 11.6	5.3 ± 1.1	50.7 ± 1.1	
Som	48.5 ± 7.7		101.3 ± 5.7	59.3 ± 10.0		67.4 ± 11.8			
Ref		33.8 ± 5.1		55.4 ± 2.3		171.0 ± 92.0		96.7 ± 68.6	

upland soils. Liming is seen to have considerably reduced the metal uptake in most of the soils especially Pb in Ystwyth, Frongoch and Parys Mt., Zn in Ystwyth and Parys Mt., and Cu in Parys Mt. is particularly marked. Cd levels are low in all experiments except Somerset where very high levels reflect those in the soil.

In the flooded experiment, Pb levels do not appear to have decreased significantly except in isolated cases, PML, HL and Ref. Zn levels however, are significantly lower in Ystwyth, Somerset, Halkyn and Ref which suggests that in these soils Zn is immobilised.

Perhaps the biggest differences can be seen for Cd - much lower levels are observed in the flooded soils, especially in the Somerset high Cd soils where uptake has decreased from 5.04 ug/g to 0.54 ug/g as a result of flooding. The effects of flooding are presented diagrammatically in Fig 6.

It is interesting to note that one of the soils showing the greatest effects of flooding is that of Somerset - which was one of the two soils where the Eh measurements indicated that redox potential was still high and reducing conditions did not prevail.

Table 16

CYPERUS ESCULENTUS STALKS - UPLAND ( $\bar{x} \pm S.D.$ )

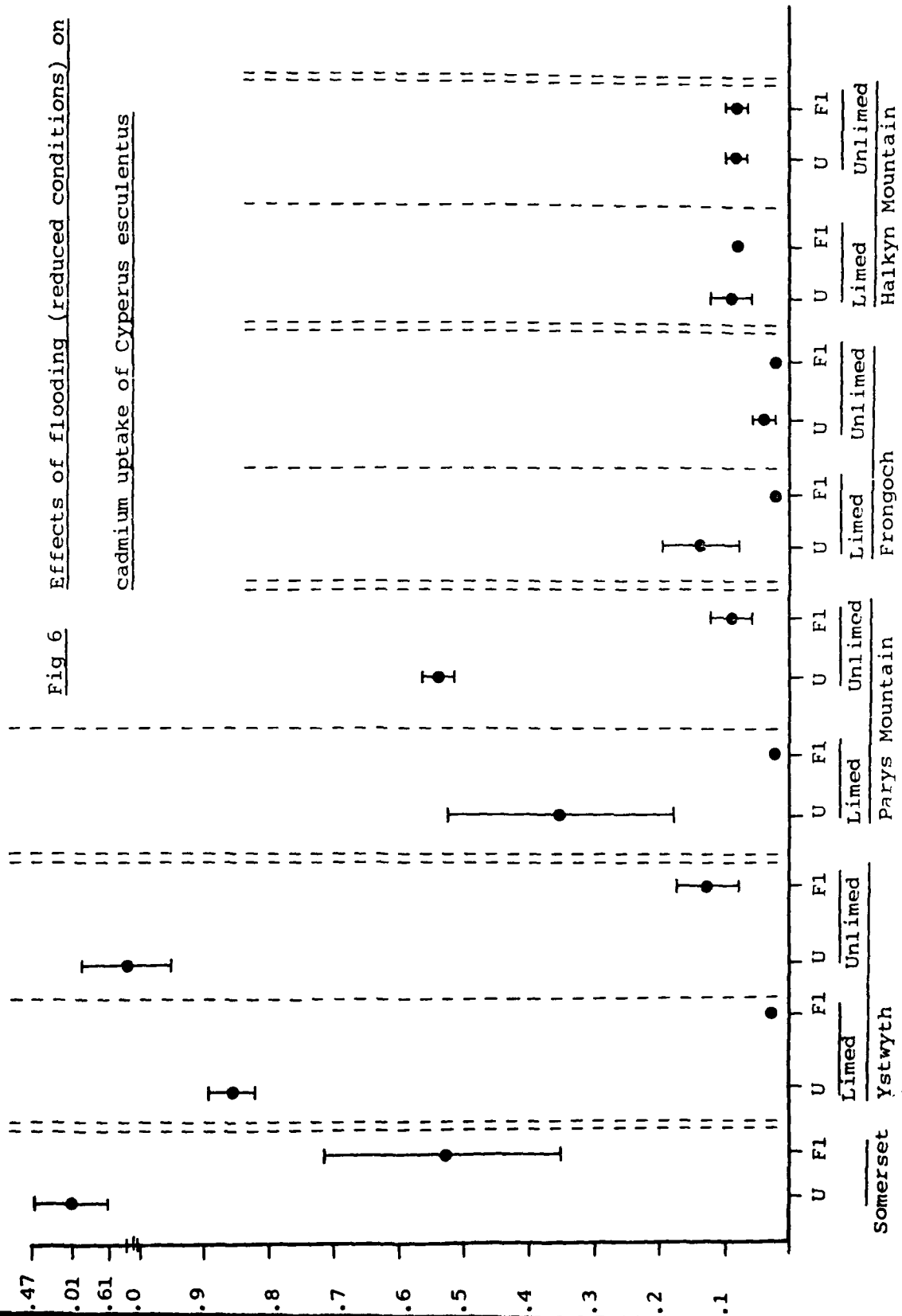
	Pb	Zn	Cu	Cd
YUL	3.6 $\pm$ 0.9	82.6 $\pm$ 35.3	2.57 $\pm$ 0.41	1.03 $\pm$ 0.12
YL	1.4 $\pm$ 0.63	24.9 $\pm$ 9.3	3.24 $\pm$ 0.7	0.87 $\pm$ 0.06
FUL	1.49 $\pm$ 0.37	11.2 $\pm$ 2.1	2.52 $\pm$ 0.26	0.04 $\pm$ 0.01
FL	0.96 $\pm$ 0.48	11.5 $\pm$ 4.2	2.66 $\pm$ 0.37	0.14 $\pm$ 0.1
S	0.94 $\pm$ 0.09	103 $\pm$ 26.5	2.68 $\pm$ 0.83	5.04 $\pm$ 0.76
PM UL	11.15 $\pm$ 1.58	38.4 $\pm$ 8.4	26.4 $\pm$ 6.87	0.55 $\pm$ 0.01
PM L	5.87 $\pm$ 2.3	16.8 $\pm$ 8.8	4.72 $\pm$ 2.1	0.36 $\pm$ 0.32
HUL	3.32 $\pm$ 1.11	77.5 $\pm$ 28.6	5.90 $\pm$ 0.63	0.08 $\pm$ 0.01
HL	2.39 $\pm$ 0.73	69.9 $\pm$ 26.5	6.01 $\pm$ 0.56	0.09 $\pm$ 0.06
Ref.	2.84 $\pm$ 2.15	6.57 $\pm$ 1.9	2.09 $\pm$ 0.8	0.10 $\pm$ 0.08

Table 17

CYPERUS ESCULENTUS STALKS - FLOODED ( $\bar{x} \pm \text{S.D.}$ )

	Pb	Zn	Cu	Cd
YUL	2.06 $\pm$ 1.08	15.7 $\pm$ 1.6	2.11 $\pm$ 0.07	0.13 $\pm$ 0.07
YL	1.62 $\pm$ 0.6	16.8 $\pm$ 2.1	2.03 $\pm$ 0.33	0.02 $\pm$ 0
FUL	1.47 $\pm$ 0.19	13.55 $\pm$ 8.15	1.71 $\pm$ 0.24	0.02 $\pm$ 0
FL	1.89 $\pm$ 0.89	7.89 $\pm$ 3.4	1.43 $\pm$ 0.11	0.01 $\pm$ 0
S	1.24 $\pm$ 0.4	81.8 $\pm$ 35.7	2.1 $\pm$ 0.97	0.54 $\pm$ 0.18
PM UL	11.62 $\pm$ 3.8	24 $\pm$ 4.0	17.5 $\pm$ 0.00	0.09 $\pm$ 0.03
PM L	2.3 $\pm$ 1.4	14.5 $\pm$ 3.1	8.4 $\pm$ 0.83	0.15 $\pm$ 0.08
HUL	7.35 $\pm$ 1.4	167 $\pm$ 42.3	20.6 $\pm$ 6.8	0.09 $\pm$ 0.01
HL	1.32 $\pm$ 0.7	122 $\pm$ 11.04	6.7 $\pm$ 0.48	0.08 $\pm$ 0.01
Ref.	0.7 $\pm$ 0.14	13 $\pm$ 1.7	1.28 $\pm$ 0.28	0.04 $\pm$ 0.00

Fig 6 Effects of flooding (reduced conditions) on

Cadmium uptake of *Cyperus esculentus*

### Spartina alterniflora

A map of the sampling sites of the estuaries is given here for reference (see Fig 7). All of the estuaries are situated on the west coast of the British Isles and some are already well documented (Avon ref). These sites were chosen from several which had been previously sampled and are detailed in Table 18.

Spartina growth in terms of both height and weight is indicated in Table 19, where it can be seen that the greenhouse experiment fared much better than that of the field. In the field also WES Spartina fared better than the local Dyfi Spartina, presumably because the local Spartina is stunted by the sandy nature of the environment, whereas the WES Spartina, in foreign mud, thrived in the finer particles of the other estuaries.

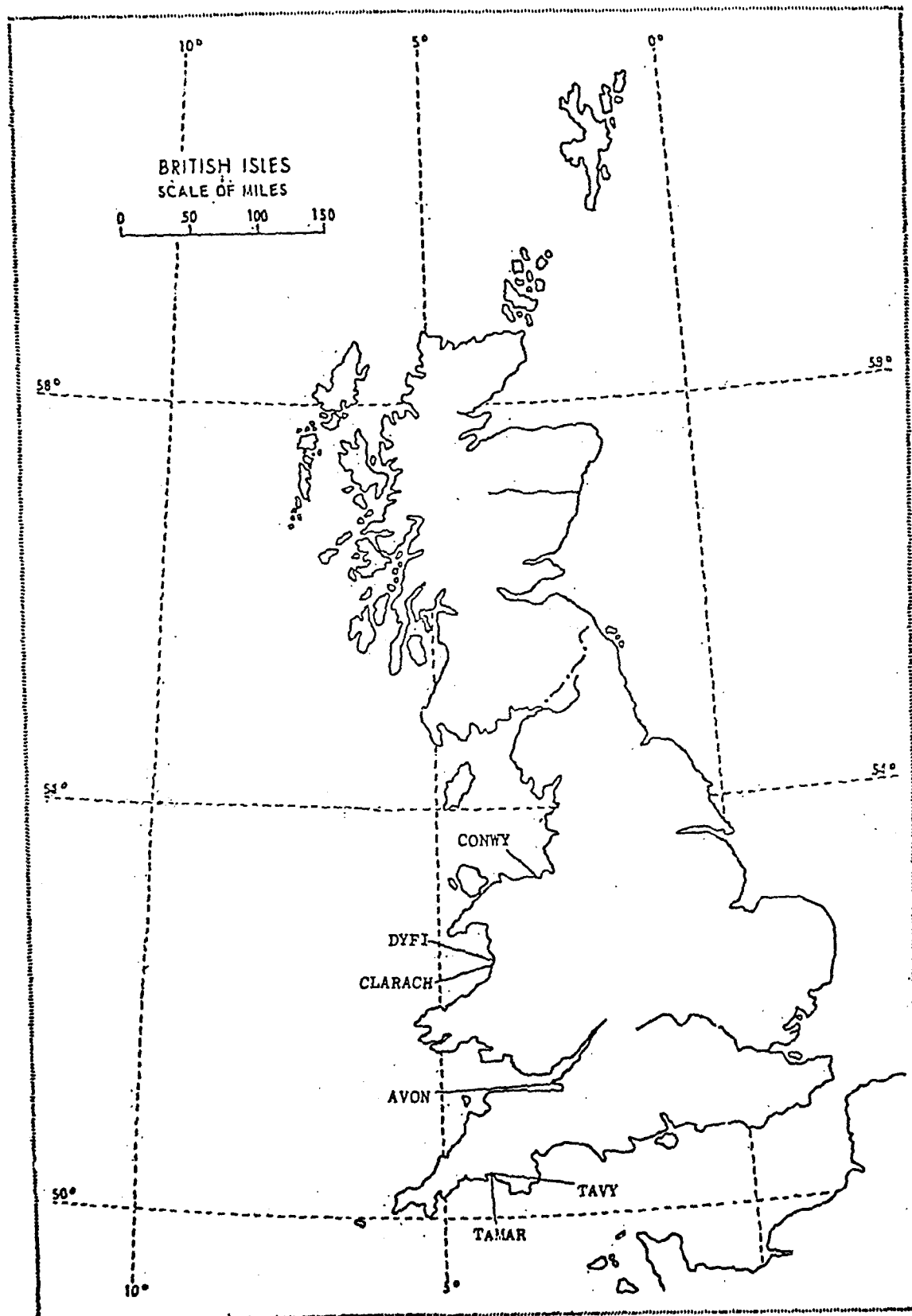
Spartina were analysed for heavy metals in the same way as the other plant material and the results are presented in Tables 20 and 21.

In general, field conditions yielded higher metal levels than greenhouse, except for Cd where little difference was observed and all levels were low.

The estuary which produced the Spartina with the highest metal levels, especially Pb and Zn was the River Clarach, secondly the River Avon, followed by the two southern estuaries, the Tamar and Tavy. In the latter two, Cu levels were slightly elevated, but not to the extent that might have been expected bearing in mind the high levels in the actual mud (see Table 18).

Figure 7

DISTRIBUTION OF SEDIMENT SAMPLING SITES





ESTUARINE SEDIMENTS - Table 8

 $\mu\text{g.g}^{-1}$  TOTAL METAL CONTENT

LOCALITY	Pb			Zn			Cu			Cd		
	$\bar{x}$	SE	CV	$\bar{x}$	SE	CV	$\bar{x}$	SE	CV	$\bar{x}$	SE	CV
AVON N.	44	3.05	9.6	105	15.5	21.0	17	0.57	5.0	0.55	0.05	12.9
*AVON NW.	43	2.6	8.1	92	4.0	6.2	17	1.8	15.7	0.52	0.04	9.6
AVON S.	28	4.9	24.9	58	0.40	0.99	5.9	3.6	87.5	0.41	0.05	15.7
*TAVY B.F.	127	9.7	10.8	180	24.6	19.4	149	2.4	2.2	0.90	0.12	18.9
TAMAR E.	165	10.4	8.9	73	2.5	4.8	73	10.8	20.8	0.52	0.10	26.1
*ST. DOMINICK	99	13.2	18.9	147	2.9	2.8	159	3.1	2.8	1.28	0.09	9.9
LLANELLI DOCK	23	7.1	42.7	35	2.7	10.6	14	4.2	41.7	0.35	0.05	18.4
LLANELLI W.	11	1.0	12.7	19	0.20	1.4	3.6	1.7	67.7	0.30	0.05	21.6
WACHYNYS	13	1.1	11.8	45	5.9	18.4	11	0.9	11.1	0.46	0.06	17.1
USK	36	6.1	24.3	65	2.6	5.6	18	2.8	21.8	0.49	0.08	23.1
LERI	118	2.8	3.3	61	3.8	8.9	13	2.3	24.2	0.43	0.06	21.6
*CLARACH	1103	23	2.9	30	0.4	1.9	67	15	31.5	0.31	0.01	2.3
CONVY U.	28	6.3	32.1	38	1.2	4.3	3.6	2.0	76.4	0.22	0	0
*CONVY L.	93	60	91.1	61	6.6	15.3	14	1.2	12.1	0.67	0.17	35.9

\* Selected.

Table 19

Spartina alterniflora - mean yield and height from greenhouse  
and field study

	Greenhouse		Field	
	$\bar{x}$ (g)	Ht (cm)	$\bar{x}$ (g)	Ht (cm)
Avon	39.3 $\pm$ 8.2	85 $\pm$ 19	21.1 $\pm$ 12.3	39 $\pm$ 14
Tavy	27.3 $\pm$ 20	79 $\pm$ 27	20.3 $\pm$ 5.7	37 $\pm$ 11
Tamar	58.9 $\pm$ 30.1	89 $\pm$ 23	33.3 $\pm$ 18.7	42 $\pm$ 12
Conwy	46.2 $\pm$ 9.3	83 $\pm$ 18.5	10.6 $\pm$ 2.1	33 $\pm$ 6
Clarach	43.6 $\pm$ 9	108 $\pm$ 6.8	11.5 $\pm$ 5.9	29 $\pm$ 8
Dyfi	24.9 $\pm$ 4.6	84 $\pm$ 13	-	-
Ref	101.5 $\pm$ 19.2	119 $\pm$ 19	16.5 $\pm$ 11.7	33 $\pm$ 9

Table 20

Heavy metals content of *Spartina alterniflora* grown in field conditions ( $\bar{x} \pm S.D.$ )

	mg/kg			
	Pb	Zn	Cu	Cd
Avon	7.02 $\pm$ 2.06	42.1 $\pm$ 7.99	4.22 $\pm$ 0.38	0.045 $\pm$ 0.02
Clarach	12.62 $\pm$ 2.86	52.21 $\pm$ 2.4	6.41 $\pm$ 0.75	0.052 $\pm$ 0.01
Conwy	7.17 $\pm$ 1.52	38.25 $\pm$ 11.5	4.04 $\pm$ 0.94	0.06 $\pm$ 0.01
Tamar	8.89 $\pm$ 2.5	30.8 $\pm$ 13.7	5.51 $\pm$ 1.64	0.051 $\pm$ 0.01
Tavy	7.28 $\pm$ 0.82	36.95 $\pm$ 1.27	6.05 $\pm$ 1.01	0.054 $\pm$ 0.01
Ref.	4.31 $\pm$ 2.03	33.06 $\pm$ 7.4	4.67 $\pm$ 1.17	0.022 $\pm$ 0.00

Table 21

Heavy metal contents of *Spartina alterniflora* grown under greenhouse conditions ( $\bar{x} \pm S.D.$ )

mg/kg				
	Pb	Zn	Cu	Cd
Avon	4.17 $\pm$ 1.23	14.95 $\pm$ 1.89	2.21 $\pm$ 0.24	0.036 $\pm$ 0.01
Clarsach	9.78 $\pm$ 2.64	32.93 $\pm$ 2.68	3.03 $\pm$ 0.61	0.05 $\pm$ 0.02
Conwy	3.94 $\pm$ 0.81	22.08 $\pm$ 4.95	1.53 $\pm$ 0.2	0.04 $\pm$ 0.00
Tamar	3.23 $\pm$ 0.75	20.3 $\pm$ 7.15	2.13 $\pm$ 0.19	0.04 $\pm$ 0.01
Tavy	4.15 $\pm$ 1.27	22.63 $\pm$ 3.77	2.09 $\pm$ 0.26	0.065 $\pm$ 0.01
Ref.	2.02 $\pm$ 0.88	24.14 $\pm$ 2.12	2.86 $\pm$ 0.52	0.035 $\pm$ 0.00
Dyfi	2.36 $\pm$ 0.09	15.74 $\pm$ 1.33	1.03 $\pm$ 0.19	0.023 $\pm$ 0.01

### Prospective

Several analyses have yet to be completed, especially the detailed analysis of the experimental estuarine mud both in field and greenhouse conditions.

Relationships between uptake and sediment will be investigated further, hence many statistical analyses are required.

The Halkyn experiment produced very strange reversed growth patterns, hence the entire experiment is being repeated over the summer of 1983. It is hoped that these responses will be explained - a new soil has been collected from the same area, and this has a far higher Pb and Zn content than the original soil used, which did not contain the elevated metal levels that we had originally hoped for.

Some additional extractions of soils and sediments are planned to allow the soil and plant data to be modelled by the GEOCHEM metal speciation prediction program.

APPENDIX 1

U.C.W., ABERYSTWYTH

GEOGRAPHY DEPARTMENT

LABORATORY TECHNICAL LEAFLET NO. 3.

### PREPARATION OF SOIL SAMPLES

On arrival at the laboratory the sample must ordinarily be registered in the departmental collection. The field number is replaced by the permanent sample number and the following details recorded in the record book : grid reference (full reference) date, abbreviated address, sample type (e.g., field or garden, sample depth) any other pertinent information and name of sampler.

If it is intended to determine an unusually labile constituent (e.g., moisture content, nitrate/ammonia nitrogen, mercury, pH of waterlogged soils etc.) the soil is analysed immediately it arrives in the laboratory. The only preparation possible will be the removal of stones and large biological debris. For all other determinations the sample is 'air-dried' before analysis. The soil is spread thinly on a clean polyethylene sheet and either allowed to dry on the bench-top at room temperature, or in an oven at approximately 25°C. In either case the soil must not be exposed to aerosol contamination or to drips from condensing moisture. It is best not to let clay soils dry out completely since they tend to harden and the subsequent grinding is made difficult.

When the soil sample is gently ground in an acid-clean porcelain pestle and mortar. Prolonged grinding should be avoided and it is not necessary to break down all aggregates. The sample is sieved through nylon mesh of 2 mm. aperture to yield 'fine earth' which is then stored in a polyethylene bag. Coarse (>2mm) material may be washed for further sieving and identification of rock type. If the coarse material is likely to exceed 10% of the total sample weight then the actual proportion should be estimated by weighing and subsequent calculations based on the fine earth should be adjusted. Thoroughly clean the pestle and mortar between samples.

The polyethylene bag containing the dried sample should be stored in numbered boxes. Close the bag in the same way as when it was filled in the field but secure with masking tape since rubber bands may perish.

### HEALTH AND SAFETY NOTE

Disposable paper masks are provided to reduce the inhalation of dust. Urban soils, especially parks, may contain the eggs etc. of harmful organisms so wash your hands carefully.

B.E.DAVIES

January 1979.

U.C.W. ABERYSTWYTH

GEOGRAPHY DEPARTMENT

LABORATORY TECHNICAL LEAFLET NO.4

THE PREPARATION OF PLANT MATERIAL

If plant samples cannot be processed immediately they are received in the laboratory they should be stored in a cool place or the refrigerator. Wash the material in distilled water to remove all visible dirt : prolonged washing may leach out constituents from the tissue. Vegetables may be scrubbed with a nylon pan scrubber or peeled. Cut the material or slice it and set to dry on aluminium foil at 80°C. Mill or grind and store in acid-clean glass bottles or plastic containers.

B.E. Davies



U.C.W., ABERYSTWYTH

GEOGRAPHY DEPARTMENT

LABORATORY TECHNICAL LEAFLET NO. 10

LABORATORY DETERMINATION OF SOIL REACTION

METHOD

Soil reaction (pH) is determined using the pH meter for which separate instructions are provided. The determination may be made following equilibration with distilled water (pH<sub>w</sub>) or 0.01M CaCl<sub>2</sub> solution (pH<sub>s</sub>).

Weigh (10g) or measure (10 ml) of soil (fine earth) into a small beaker (50 or 100 cc) and add water or calcium chloride solution (25 cc). The resulting liquid level must be above the tiny wick in the side of the combined electrode. Stir and allow to stand. Stir again after 15 minutes, then allow to stand for a further 15 minutes, stir, and measure the pH of the suspension immediately.

The pH meter is not normally switched off, but if it has been, allow at least 15 minutes for it to warm up. The meter will need adjusting using standard buffer solutions prepared from buffer tablets. For soil determinations, solutions of pH 4.0 and 7.0 are used. Since the measurement is temperature dependent either measure the temperature of your suspensions and adjust the meter accordingly, or, use the temperature compensating probe. Check that the tube of the electrode is filled with saturated potassium chloride solution. The cap on the side of the electrode is removed during measurements and replaced afterwards. The electrode is left standing in distilled water.

After each measurement thoroughly rinse the electrode and temperature probe (if used) with distilled water. Before the next measurement touch a piece of filter paper to the electrode to remove surplus water. When inserting the electrode into the soil suspension, swirl the beaker VERY GENTLY so as to displace the film of water on the electrode.

REAGENT

A solution approximately 0.01M of CaCl<sub>2</sub> is made by dissolving 1.30g of AR CaCl<sub>2</sub> in 1000 ml of distilled water.

INTERPRETATION

	<u>Water</u>	<u>CaCl<sub>2</sub></u>
Strongly acid	<4.5	<4.0
Moderately acid	4.5 - 5.5	4.0 - 5.0
Slightly acid	5.5 - 6.5	5.0 - 6.0
Neutral	6.5 - 7.5	6.0 - 7.0
	<7.5	<7.0

(DAVIES, B.E. (1971). Soil Sci. Soc. Amer. Proc. 35, 551-2).

B.E.DAVIES

January, 1979.

U.C.W., ABERYSTWYTH

GEOGRAPHY DEPARTMENT

LABORATORY TECHNICAL LEAFLET NO. 8

THE GRAVIMETRIC DETERMINATION OF SOIL ORGANIC MATTER

Organic contents of soils may be determined by several chemical methods, instructions for which are given in SOIL CHEMICAL ANALYSIS by M.L. Jackson and Technical Leaflet No. 9. A close approximation to the organic matter content is obtained by measuring the loss in weight when a sample of the soil is ignited. This loss in weight is greater than the organic matter content because it includes losses of other constituents, especially the water bound inside minerals.

METHOD

Put approximately 10 g of dry soil (<2 mm fine earth fraction) into a labelled crucible of known weight. Weigh the crucible plus soil and place in the oven at 100°C for 24 hours; reweigh and then place in the electric furnace; ignite at 430°C for 24 hours. Allow to cool in a desiccator and reweigh. Express the 'Loss on Ignition' as a percentage of the dry soil. Make all weighings on an analytical balance. Results may be computed using the program BESHAM (in BASIC) which is available on the Department's computer terminal. BEPRECIS is available for the precision of duplicates.

REFERENCES

- MILL, D.F. J. Soil Science, 1964, 15, 84-92.  
DAVIES, B.E. Soil Sci. Soc. Amer. Proc., 1974, 38, 150-151.  
JACKSON, M.L. Soil Chemical Analysis, 1962, Constable & Co.

B.E.DAVIES

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LABORATORY TECHNICAL LEAFLET NO. 19

PARTICLE SIZE ANALYSIS OF SOIL : HYDROMETER METHOD

Laboratory Technical Leaflet No. 18 referring to the pipette method of analysis and L.T.L. No. 16 on dry sieving techniques should also be consulted.

Method

Weigh the air dry fine earth fraction of the soil (normally 50g., but 100g. if very sandy) into a tall 800 ml. beaker; add hydrogen peroxide solution (60 ml. of 20 vol.  $H_2O_2$ ) and warm until the copious frothing ceases. If necessary add further peroxide solution and warm. When all frothing has finished, boil for a few minutes to destroy excess peroxide solution. Carbonates may be destroyed by HCl (vide No. 18).

Add sodium hexametaphosphate ("Calgon") solution (10 ml. of 5% (W.V.) solution) and transfer the contents of the beaker to the mechanical stirrer. Stir for 10 minutes. Transfer the suspension to the specially graduated cylinder and fill to the lower mark with water with the hydrometer in place (if you used 100g. soil, fill to the upper mark). With a one-litre measuring cylinder fill to the 1000 ml. mark without the hydrometer in place.

Remove the hydrometer, cover the end of the cylinder with the flat palm of your hand, and thoroughly mix by inverting the cylinder several times. Place the cylinder down on the bench and immediately introduce the hydrometer gently into the suspension simultaneously starting the stop clock. Cling film over the mouth of the cylinder will give added security if applied when dry. An alternative method of agitation using the perforated 'Horlicks' plunger can be used if necessary. Complete agitation is essential in this method.

Hydrometer measurements at 40 seconds (finer than 50  $\mu$ m), 4 minutes (finer than 20  $\mu$ m) and 2 hours (finer than 2  $\mu$ m) are made.

The hydrometer is introduced about 20 seconds before the measurement time, lowered gently to a position of approximately neutral buoyancy and allowed to settle. The reading on the scale 0 to 60 grams (per litre) is read (to the nearest 0.5 gram) from the top of the meniscus. The 40 seconds and 4 minute readings may be repeated after reagitation to ensure reliable results. The hydrometer should not be left in the suspension between readings as sediment can settle on its shoulders and weigh it down. Great care in handling the hydrometers (£7 each!) should always be taken. Note the suspension temperature.

After the 2 hour (clay) reading has been made pour off the supernatant and transfer to the tall 800 ml. beakers and mark at 10 cm. from the base. Make up to this mark with water, measure the temperature, stir and allow to stand for approximately 5 minutes (the exact time is derived from the table for "silt and clay" in the instruction sheet for the pipette method); then, pour away the supernatant. Repeat until the supernatant is clear at the end of the standing time: only the sand fractions are now present in the beaker. Dry in the oven (100°C) and separate the coarse and fine fractions using an appropriate sieve. Weigh these fractions.

Calculation of results:-

The FINE SAND and COARSE SAND fractions are determined directly by expressing their weights as %.

The hydrometer graduations refer to a temperature at 19.4°C. For every 1°C ABOVE 19.4°C ADD 0.3 to the reading, while for every 1°C BELOW 19.4°C SUBTRACT 0.3. A further correction must be made to eliminate the effect of the "Calgon" solution: subtract 0.5 from the corrected readings. The resulting figure is the true value, as N grams/litre of material in suspension.

Hence  $\frac{N}{\text{wt. soil}} \times 100 = \% \text{ of the particular fraction.}$

The reading at 4 minutes gives the % (CLAY + SILT) and that at 2 hours % CLAY only: % SILT is found by difference. These data refer to the I.S.S.S. size limits. (The reading at 40 seconds gives the % (CLAY + SILT) using the U.S.D.A. size limits of 50  $\mu\text{m}$ ).

Ref.

BOUYCOUCOS, G.J. Agronomy Journal, 1951, vol. 43, 434-438.

B.E. Davies/I.M.S. Laidlaw  
October, 1980

U.C.W. ABERYSTWYTH

GEOGRAPHY DEPARTMENT

LABORATORY TECHNICAL LEAFLET NO. 13.

SOIL EXTRACTION : HEAVY METALS

This method extracts approximately 100% total Zn, 80% total Pb and 60-80% total copper from the soil.

Weigh approximately 5g fine earth into a labelled and weighed 50 ml beaker. Put to dry at 100°C for 24 hours; reweigh; ignite at 430°C for 24 hours, cool, reweigh. Record all weights (W1, W2, W3, W4).

To the soil add concentrated nitric acid (20 cc), cover with a glass and heat on the hotplate (110°C) for 30 minutes. Remove and rinse cover glass and take the contents to near dryness (120°C). The soil is "dry" when it does not move on tilting the beaker but the soil surface may gleam slightly : baking MUST be avoided. Repeat this procedure once.

To the dry soil slowly add 0.01 M  $\text{HNO}_3$  (normally just < 50 ml but just < 25 ml if the total Pb is < 20 ppm); break up the soil and stir with a glass rod; rinse the rod with the last few mls of acid. Set to warm on the hotplate (50°C) for 15 minutes, then filter through a fluted Whatman no. 540 paper into 25 ml volumetric flasks. Make up to the mark with distilled water.

SAFETY NOTE

In all operations involving concentrated nitric acid, wear safety goggles and gloves. Do not wear sandals or "open" footwear. Wash acid splashes immediately with copious cold water.

CALCULATION

Moisture content should be expressed as % oven dry weight. The loss after ignition is a measure of the soil's organic content and should also be expressed as % oven dry weight. A computer program for carrying this out is available.

B. F. DAVIES

U.C.W., ABERYSTWYTH

GEOGRAPHY DEPARTMENT

LABORATORY TECHNICAL LEAFLET NO. 7

THE DRY ASHING OF PLANT MATERIAL

Refer to leaflet No. 4 for instructions concerning initial preparation and milling of plant material.

Weigh between 1 and 5 g of oven-dried, ground material into a clean, dry weighed tall-form Pyrex glass beaker (50 cm<sup>3</sup> capacity) and reweigh.

Put the beaker in an electric-furnace, placing well back, and between the sample beaker(s) and the door arrange two rows of empty beakers close together. This prevents the draught from around the door cooling the samples.

Set the temperature control to 400°C and switch on; when the set temperature is reached increase gradually to 500°C and maintain for 24 hours. The resulting ash should be white or pale grey.

Cool and reweigh : remember the ash is light and readily lost by blowing. Work in the fume cupboard. Dampen the ash with distilled water and carefully add concentrated AR HNO<sub>3</sub> (10-15 cm<sup>3</sup>) drop by drop guarding against loss of material during effervescence.

Warm on a hot plate and take to dryness. Either, dissolve the residue in 20 cm<sup>3</sup> 0.01M HNO<sub>3</sub> and filter into any convenient container (e.g., a clean Erlenmeyer flask); or dissolve the residue in a convenient volume of 0.01M HNO<sub>3</sub> and filter into a volumetric flask, wash the beaker, filter paper and funnel with acid and make to volume.

The extract is ready for analysis and the dry and ash weights and % ash are calculated.

SAFETY NOTE

Remember to wear protective clothing when handling acids; splashes should be washed away IMMEDIATELY with copious water. Concentrated nitric acid is a strong oxidising agent.

B.E. Davies  
(Rev'd February 1983)

END

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